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Characterization and Nanostructured Enhancement of Boiling Incipience in Capillary-Fed, Ultra-Thin Sintered Powder Wicks

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ABSTRACT

Next-generation thermal management applications will require passive heat spreading at a lower thermal resistance, higher dryout tolerance, and with thinner profile devices than current vapor chambers. Such performance improvements may be achieved by augmenting evaporation and boiling heat transfer via patterning the internal wick or nanostructuring the wick surface in the region of heat input. Test samples composed of 200 µm thick sintered copper powder layers are investigated because they can be integrated into vapor chambers with an overall thickness of 1 mm. Carbon nanotubes (CNTs) are grown onto patterned and monolithic samples by a microwave plasma chemical vapor deposition synthesis technique, and are functionalized to ensure high wettability with the test fluid, water. Performance of the test samples is evaluated in an experimental facility which replicates the heat input and capillary fluid-feeding mechanisms at the evaporator section of a vapor chamber. High-speed visualizations are performed to identify the vapor formation regimes. Monolithic samples are shown to dissipate heat fluxes greater than 400 W/cm² over 0.25 cm² prior to dryout. A noteworthy heat transfer enhancement mechanism observed is reduction of the required superheat for boiling incipience by addition of a CNT coating. Predictable transition from the evaporation to boiling regimes at a lowered superheat is critical due to the lower thermal resistance associated with boiling. Multiple repeated tests on identically prepared samples reveal that the CNT coating reduces the average incipience substrate superheat by 5.6 °C compared to uncoated samples.

Keywords: vapor chamber, heat pipe, carbon nanotube (CNT), evaporation, boiling incipience, thermal resistance

NOMENCLATURE

Greek symbols

\( \delta \)  limiting thermal boundary thickness, m
\( \rho \)  density, kg/m³
\( \sigma \)  surface tension, N/m

Subscripts

Cu  copper property
ref  vapor reference temperature
s  solder
sat  saturation temperature

INTRODUCTION

The ever-increasing heat dissipation from next-generation consumer, automotive and military electronics devices will require significant advancements over conventional thermal management strategies. The simultaneous decreases in available packaging size add to the challenge. For many applications with exacting space and weight constraints, liquid cooling approaches with active pumping are not viable. Passive phase-change cooling approaches, such as vapor chamber heat spreaders, are widely utilized due to their superior thermal performance, high reliability, low cost, and simplicity of operation; however, their use in such applications has been restricted in the past due to limitations on the maximum supported heat transfer fluxes and rates.

Ultra-thin, passive phase-change devices can serve as a so-called “thermal ground plane” and can be substituted into numerous applications that currently rely on solid conduction heat spreaders, thereby reducing the thermal resistance to the ambient without any perceptible change to the overall packaging strategy. An improved understanding and enhancement of the boiling process in thin porous structures fed by capillary action is required in order to realize vapor chambers that dissipate hundreds of watts per square centimeter over small areas and short distances. High wall superheats are likely to be reached well before capillary dryout at such form factors, which causes the onset of boiling during operation in contrast to the behavior in typical heat pipes that transport lower heat densities over longer distances. Several recent studies have developed experimental facilities which can provide a heat flux-controlled boiling curve under conditions which replicate the capillary fluid feeding as observed in a vapor chamber. These studies have helped...
understand performance trends for sintered screen [1,2] and sintered powder [3] layers as a function of wick parameters.

In addition to understanding the parametric trends and vapor formation regimes, several methods for augmenting performance have been investigated. One such method is patterning of the wick structure in order to incorporate large pores into the wick design, which facilitate easier vapor exit from the wick under vigorous boiling conditions. This enhancement mechanism has been attributed for increases in the dryout heat flux by as much as 350% observed by including 500 µm-wide parallel grooves into a sintered powder wick [4]; similarly, the surface superheat in the boiling regime has been reduced by up to 72% for a grid of 1 mm square recesses formed into a sintered powder wick [5]. Semenic and Catton [6] evaluated biporous wicks (having two characteristic pore sizes) and showed that the maximum dryout heat flux is increased compared to monoporous wicks due to vapor vents that form in the larger pores of the structure. However, the wick structures that exhibit this enhancement mechanism in all of the aforementioned studies are 1 to 4 mm in total thickness, and are therefore not suitable for incorporation into ultra-thin vapor chambers with total thicknesses of 1 mm or less.

Another enhancement strategy that deserves further investigation is the incorporation of nanostructures, in particular carbon nanotubes (CNTs), into the wick structures. Current research [7,8] is focused on evaluating the use of CNTs as evaporation/boiling enhancement surfaces in an attempt to exploit their high intrinsic thermal conductivity [9-12] and tunable nanoporous structure. Pool boiling tests performed with highly wetting fluorinated liquids have shown that coating smooth surfaces with a CNT array increases the critical heat flux (CHF) [13-15] and reduces the superheat required for boiling incipience [15,16]. However, because CNTs are naturally hydrophobic [17], they must be functionalized for use with water, which is the most effective heat transfer fluid available for use in a vapor chamber for electronics cooling applications.

Several functionalization techniques that render CNTs hydrophilic for investigation of capillary-fed boiling using water as the working fluid have been demonstrated. Cai and Chen [18] performed a hydrochloric acid treatment on a CNT biwick composed of alternating 100 µm wide CNT array stripes and 50 µm wide exposed substrate grooves, and were able to sustain heat fluxes of up to 400 W/cm² at a superheat of 35 °C. Hashimoto et al. [19] exposed a 1.5 mm thick, CNT-coated sintered powder sample located inside a vapor chamber prototype to ultraviolet radiation and found it to be capable of dissipating 500 W/cm². Lastly, post-growth deposition of copper over the nanotubes has been previously demonstrated [20] by the current authors. A copper coating has the distinct advantage of being a widely used material within vapor chambers which offers proven long-term reliability and compatibility with the working fluid.

In a recent study [5], the authors obtained boiling curves for 1 mm thick patterned and CNT-coated sintered powder layers in a capillary-fed boiling test facility. Patterning of the wick was shown to increase the permeability to exiting vapor and improve performance; however, CNT-coated areas of the test surface remained flooded during much of the testing and proved to have negligible performance benefit. The current work expands the investigation to 200 µm thick sintered powder layers for use in ultra-thin vapor chambers and aims to understand the isolated and combined effects of the patterning and the functionalized CNT-coating. The enhancement mechanisms would be expected to be altered for the thinner wicks which impose less resistance to exiting vapor. Also, the superheat temperature required for boiling incipience becomes a critical design parameter in thin wick structures, and therefore, a detailed investigation is performed to quantify and predict this superheat value.

Fig. 1 illustrates a schematic diagram of an internal transport processes for a vapor chamber heat spreader with micropatterned and nanostructured surfaces.

**SAMPLE FABRICATION**

Four different wick samples are fabricated to study the heat transfer enhancement provided by the proposed augmentation features, as shown in Fig. 2. A monolithic uncoated sintered powder sample serves as the baseline for comparison against: a monolithic CNT-coated sample, an uncoated grid patterned sample, and a sample having both proposed augmentation features. Additionally, six nominally identical uncoated monolithic samples and two nominally identical CNT-coated monolithic samples are fabricated for detailed investigation of the required superheat for boiling incipience. The fabrication procedure, involving sintering of copper powder, CNT growth and copper functionalization, is maintained consistent and identical for all the samples. Furthermore, the fabrication and test procedures are identical to those used in [5], allowing direct comparison to these previous results.

**Sintered powder wick fabrication**

The copper powder microwick is sintered and attached to a solid substrate that represents the vapor chamber wall. The 25.4 mm × 25.4 mm × 0.381 mm solid substrates are composed of laminated 13%-74%-13% copper-molybdenum-
copper (Cu/Mo/Cu) sheets. This material is chosen for direct mounting of vapor chambers on electronic devices because of the matched thermal expansion coefficient with silicon. The porous wicks are fabricated at Thermacore, Inc., by placing copper particles into a mold of the desired pattern and exposing to a proprietary high-temperature forming gas atmosphere to sinter the particles. The resulting structure has a volumetric porosity of 50%. Particles of 100 µm average diameter are used for all samples. Analytical correlations predict an approximate pore radius of 21 µm and a conductivity of wicks fabricated by this process. Such analytical correlations tend to over-predict the thermal conductivity of such sintered wicks (produced using the same fabrication process) without resorting to approximate correlations. This characterization technique revealed that such analytical correlations tend to over-predict the permeability and are inadequate for determining the thermal conductivity of wicks fabricated by this process.

The samples are then functionalized by coating the CNTs with a 250 nm nominal thickness layer of evaporated copper via physical vapor deposition, making the CNT surface hydrophilic. Nominal thickness is defined as the layer thickness deposited on a flat plate, and is not the actual thickness of the copper coating on the nanotubes. Based on the nanotube diameters observed in SEM images before and after copper deposition for a preliminary sample, the actual copper coating thickness for samples used in this work is on the order of tens of nanometers.

Carbon nanotube synthesis and functionalization
For the samples coated with CNTs, a trilayer metal catalyst comprised of 30 nm of Ti, 7.5 nm of Al, and 5 nm of Fe is deposited directly on the sintered copper samples using a Varian e-beam deposition system. The Fe provides nucleation and active catalytic growth sites for the CNTs. The CNTs were grown on the catalyzed samples in a SEKI AX5200s microwave chemical vapor deposition (MPCVD) system. The system allows independent control of substrate temperature, substrate position, gas flow rates, chamber pressure, bias voltage and microwave power. The ability to control these independent parameters before and during the growth process leads to the controlled synthesis of CNTs. Details of the CNT growth procedure are provided in prior work [23]. In brief, once the sample is introduced, the system is pumped down to 2 Torr. The temperature is ramped to 900 °C under 10 Torr of N₂ for annealing, and growth is initiated under 50 sccm of H₂ and 10 sccm of CH₄. After CNT growth for a period of 10 minutes, the system is allowed to cool, and the sample is removed.

Observations of the resulting CNT growth for both patterned and monolithic samples are made via scanning electron microscopy (SEM). Fig. 4a presents a low-magnification image of the CNT-coated patterned sample that confirms consistent CNT coverage wherever the trilayer catalyst was deposited. Inset images highlight the two different regions of CNT morphology resulting from growth on the exposed substrate (Fig. 4b) and growth over sintered copper particles (Fig. 4c), at varying magnification.

Growth over the copper substrate (Fig. 4 b1) forms a mat of CNTs that conforms to the microscale surface roughness. Inspection of the sintered particles at medium magnification (Fig. 4 c1) shows that the top layer of individual particles is coated with CNTs. The line-of-sight catalyst deposition process likely prevents uniform CNT coating around the entire underside of the particles; nonetheless, CNT growth is observed on the vertical sides of the particles and penetrates into the deeper layers within the sintered powder structure.

The CNT array structure is composed of random, non-aligned nanotubes that form a matrix of permeable nanoporous cavities. The formation of CNT nanopores over the exposed
substrate (Fig. 4 b2) is expected to form a thin film of liquid and allow the base of the grid pattern to serve as a wicking surface in order to improve evaporation/boiling performance compared to the uncoated patterned sample. CNT-coating of the sintered powder surfaces (Fig. 4 c2) may alter the nucleation characteristics of the wick pores and performance in the boiling regime.

Lastly, the high magnification images in Fig. 4 b3 and Fig. 4 c3 illustrate the effectiveness of the copper functionalization technique. Copper coats each nanotube around the entire tube diameter resulting in a hydrophilic porous structure that can behave as a capillary wick. The diameter of the nanotubes is estimated from these images to be on the order of 100 nm.

TEST FACILITY AND PROCEDURE

All of the boiling curves presented in this study were generated using the experimental apparatus and procedures described below. The facility was developed to measure the substrate temperature of a porous wick material subjected to varying heat fluxes as water is fed by capillary action over the sample surface, mimicking the evaporation/boiling mechanisms and boundary conditions that would occur in a vapor chamber [3].

Prior to testing, all samples are exposed to a hydrogen plasma in the MPCVD chamber under a plasma power of 300 W with a substrate temperature at 100 °C for 10 minutes to remove any minor copper oxidation and preserve the surface wetting characteristics. In order to supply a high heat flux input to the back of the evaporator substrate, each sample is soldered to a copper heater block with a heat source area of 5 mm × 5 mm. The heater block is encased in a ceramic insulating shell. The 0.102 mm thick Pb-Sn (conductivity, $k$, 50 W/mK) solder joint is formed in an inert argon environment at 200 °C to prevent surface oxidation. The copper heater block and sample assembly is sealed vertically against the test chamber (Fig. 5a). The test chamber contains a bath with a liquid surface level fixed precisely 8.25 mm beneath the heat input area, thereby forcing the liquid to wick over the evaporator to cool the surface. The liquid water bath and vapor space in the chamber are held at the saturation temperature (100 °C) at atmospheric pressure, as monitored by internal thermocouples and a pressure transducer. A rigid borescope is sealed through the chamber wall and is attached to a Photron Fastcam-X 1024 PCI high-speed camera to allow for in situ visualization of the sample surface during testing at up to 10,000 frames per second. This apparatus can be used to view normally and at a 45° angle to the sample surface, allowing the vapor formation regimes (i.e., evaporation versus boiling) to be identified during testing as discussed in the results section.

To begin an experiment, the water in the chamber is first boiled vigorously with the Graham condenser valve open in order to purge all non-condensable gases and produce a saturated water vapor environment. For each heat flux test point, the required electrical input to the embedded cartridge heaters is provided and the system is allowed to stabilize until the substrate temperature varies by less than 0.1 °C/min for a period of 10 minutes, defined as steady state. Once steady state is reached, the high-speed camera is used to record the corresponding evaporation or boiling process for every heat flux data point shown on the boiling curve.
Each heat flux versus temperature point on the boiling curve is generated from time-averaging 5 minutes of steady-state data. The temperature drop along an array of thermocouples \( (T_r \text{ to } T_f) \) embedded along the centerline of the copper heater block (Fig. 5b) is used to determine the heat flux \( q'' \) according to Fourier’s law for one-dimensional heat flow. This calculation method allows a determination of the actual heat flux, and accounts for heat losses from the heater block insulation.

The substrate temperature \( T_{\text{substrate}} \) is found by extrapolating the measured temperatures to the substrate and accounting for the temperature drop across the solder joint according to

\[
T_{\text{substrate}} = T_4 - q'' \left[ \frac{(x_s - x_d)}{k_{Cu}} + \frac{t_x}{k_s} \right]
\]

(1)

where the copper conductivity \( k_{Cu} \) is taken as 400 W/mK. An uncertainty analysis [24] is used to predict the uncertainties in heat flux and substrate temperature; inputs to the uncertainty analysis include the following contributions: ± 0.3 °C in \( t_x \), ± 25% in \( t_s \), ± 5% in \( k_s \), and ± 1x10^{-4} mm² in \( A_s \).

**RESULTS AND DISCUSSION**

**Patterning and CNT-coating enhancement**

This section discusses the boiling curves obtained for the four sample types described above and pictured in Fig. 2. For this set of samples, the input heat flux is continually increased until dryout is reached and an associated sharp temperature rise is observed. Finer heat flux increments are used around transition from evaporation to boiling to improve resolution of this regime change within the boiling curve. The boiling curves for all samples are shown in Fig. 6. Bars of uncertainty less than ± 0.5 °C and ± 5 W/cm² in \( \Delta T \) and \( q'' \), respectively, are omitted. The “X” marks at the end of each curve indicate that the next test point resulted in total dryout of the wick structure.

The uncoated monolithic sample serves as a baseline for comparison. The boiling curve of this sample has three distinct regimes. At low heat fluxes, below 48 W/cm², liquid completely saturates the pores and heat transfer occurs via evaporation from the menisci at the top of the wick. At a substrate superheat of 8 °C, a sharp temperature drop corresponding to the onset of boiling in the wick structure is observed. This performance increase can be attributed to transition from evaporation at the top particle layer to bubble nucleation at the base of the wick, consequently bypassing conduction resistance through the saturated wick layer, as explained in [3]. The boiling curve continues at an increased slope – corresponding to an increased heat transfer coefficient – until partial dryout of the wick begins at 342 W/cm². The performance then degrades until total dryout is observed and testing is ceased. Compared to a 1 mm thick wick [5], the substrate superheat is significantly reduced in the boiling regime for the 200 µm wick; however, the heat flux supported at dryout is reduced. Based on these experimental results, it is considered critical to develop analytical or empirical predictions for the two-phase pressure drop-induced dryout heat flux for sub-millimeter thick wicks. Single-phase liquid pressure drop calculations using the estimated wick permeability would over-predict the dryout heat flux [25].

The performance of the CNT-coated monolithic wick is similar to that of the baseline sample throughout the boiling regime. A slightly higher surface superheat (~2 °C) is observed from 250 to 350 W/cm², but this difference is within the measurement uncertainty. Dryout also occurs at approximately the same heat flux. The noteworthy performance enhancement observed is that transition to the boiling regime occurs at a lower heat flux, leading to a lower thermal resistance at heat fluxes below ~60 W/cm². The significance of this enhancement should not be overlooked since many applications that may see peak heat fluxes well above 60 W/cm² actually operate at lower heat fluxes a majority of the time. Due to the importance of predicting this transition, a detailed investigation of the substrate superheat required for boiling incipience is conducted as discussed later in this work.

![Fig. 6 Boiling curves comparing 200 µm thick patterned and CNT-coated sintered copper powder samples to a baseline uncoated monolithic sample.](image)
exit through the open grid areas, as was observed for thicker wicks [5].

Lastly, the CNT-coated grid-patterned sample can demonstrate whether or not CNTs arrays grown directly on the substrate in the patterned recesses can actively wick fluid and enhance boiling performance. As shown in the boiling curve in Fig. 6, at heat fluxes below 250 W/cm², the CNT-coated patterned sample has a higher thermal resistance than the uncoated version. Visualization of the boiling processes for both surfaces does not reveal a difference in the vapor formation behavior that can explain this slight performance reduction. However at ~250 W/cm², the boiling curves intersect and the dryout heat flux for the CNT-coated sample is subsequently extended by 28%. Visualization of the CNT-coated sample surface at this point of crossover (Fig. 7b) reveals that the CNTs within the grid patterns are wetted and the liquid front is observed to actively recede and re-wet during intense evaporation. Compared to the uncoated sample at the same heat input, evaporation from these grid areas is the mechanism which delays dryout. While in the current study, the 200 µm thick wicks do not benefit from patterning, at some intermediate wick thickness, it is viable that a CNT-coating may serve to improve dryout tolerance while the patterned recesses serve to lower the thermal resistance by increasing the permeability to exiting vapor.

**Boiling incipience characterization**

The experimental results above reveal the importance of understanding transition from the evaporative to the boiling regime during capillary-fed boiling in the design of vapor chambers subjected to high heat fluxes. It is desirable to operate at a reduced thermal resistance in the boiling regime and to avoid repeated fast thermal transients caused by incipience of boiling at large superheats that may damage sensitive electronics. Therefore, any proposed performance enhancements must improve predictability and reduce the superheat at which incipience occurs. Similar to the results above, a large superheat at incipience transition has been shown in device-level testing [26] for monolithic sintered power samples. However, in actual devices, the saturation pressure continuously varies with heat input and is not precisely known at the evaporator based on measured wall temperatures alone. By testing at a single saturation pressure, as performed in the current study, a clean empirical prediction can be made for a specific operating pressure. The results can then be extrapolated to other saturation pressures based on a nucleation parameter that predicts the relative required incipience superheat for a fluid, based on thermophysical properties [27]:

\[
N = \frac{\sigma_f T_{sat}}{\rho_g h_{fg}}, \quad \frac{N_{p_{sat},1}}{N_{p_{sat},2}} \approx \frac{\Delta T_{onset,1}}{\Delta T_{onset,2}}
\]

The required incipience superheat can then be used in models [28] that require empirical inputs to predict device performance at varying saturation pressure.

Due to the inherent variability in boiling incipience, one approach to determining the required superheat is to repeat tests with identical surfaces and develop a probabilistic representation of boiling characteristics. This approach has been previously used to predict pool boiling incipience for highly wetting fluids that are prone to large temperature overshoots [29,30]. In the current study, a similar approach is used, but is instead applied to capillary-fed boiling from sintered powder structures using water and to evaluate the efficacy of CNTs to reduce incipience overshoot. As mentioned in connection with the sample fabrication details above, six uncoated and two CNT-coated additional monolithic samples were prepared for this repeated testing. Multiple samples are used to account for sample-to-sample variability as well as variation while testing the same sample over multiple trials.
At least three boiling curves are obtained for each sample by starting at ~20 W/cm² and increasing the heat flux in 5 W/cm² increments until boiling is visualized at steady state (and at least to above 60 W/cm²) and testing is ceased. A boiling curve for a single trial is shown in Fig. 8 and depicts the process used to extract the incipience superheat from the transient data. First, the heat input is increased and allowed to stabilize (at point “1”). Following the next input power step, the temperature slowly rises until a sudden transient temperature drop (see inset plot) occurs concurrently with boiling incipience, before the system again stabilizes (at point “3”). The required incipience superheat is estimated to be the temperature immediately prior to the transient temperature drop (point “2”) and is indicated by a red dashed line on the boiling curve. Although rigorous care was taken to limit sample oxidation and to ensure that each test run was identical, some degree of unavoidable surface aging was observed between trials, typical of such testing [29]. In all cases, the aging led to an increased surface temperature between the first and second trials. No further surface aging was observed between subsequent test runs and, therefore, an aging protocol to discard the first trial was adopted.

The compiled boiling curves for all of the uncoated and CNT-coated samples are shown in Fig. 9. The point of incipience for each trial is circled. To facilitate comparison with results from the literature, the saturation temperature is used as the reference temperature on the x-axis. However, as discussed in the experimental facility description, the precise sample surface temperature is not known due to spreading in the substrate. Therefore, the substrate temperature is used instead to determine the superheat. As a first-order approximation to understand the implication on the results shown, the assumption of one-dimensional heat flow through the substrate predicts a temperature drop of 0.3 to 1.1 °C between the substrate and the surface temperature of the wick, in the range of heat fluxes from 20 to 80 W/cm². Horizontal uncertainty bars are not included on the graph. The uncertainty in ΔT increases from ± 0.4 to ± 0.6 °C from 20 to 80 W/cm².

Uncoated monolithic samples. Boiling curves for 18 test runs using the uncoated samples are shown in Fig. 9a. The substrate superheat at incipience has a wide range from 2.1 to 19.8 °C with a mean value of 9.3 °C and standard deviation of 6.5 °C. The onset of boiling was apparently random and no trends appeared with respect to the number of trials performed or between samples. While there is some spread in steady-state performance within the evaporation and boiling regimes, this is not indicative of sample aging. For a given sample, the boiling curves from multiple trials overlapped closely, except for the random superheat at which incipience occurred. This suggests that for nominally identical monolithic samples, the performance may vary slightly between samples. This behavior is not surprising for the 200 µm thick wicks considered in this work that are only a few particle layers deep. Small variations in the random formation of particles during sintering could cause noticeable changes in the structure and boiling performance. A slight increase in the evaporation regime substrate superheat with increasing heat flux was also observed. This is more clearly described in Fig. 10 which shows all steady-state data points identified by regime. The dashed lines in this plot are meant only to highlight the general trends. The drastic performance difference between the two regimes reiterates the motivation for operating in the boiling regime at lowered heat fluxes.

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It is well documented that large incipience superheats are observed for pool boiling from smooth surfaces using highly wetting fluids due to the vapor embryo entrapment process. Surface superheats for pool boiling of FC-72 have been observed in the range of 20 - 40 °C from platinum surfaces [29] and 10 - 20 °C from copper surfaces [30]. When spherical copper contacts were pressed against the surface, the incipience superheat was reduced to less than 10 °C through creation of high cavity angle sites for vapor embryo trapping [30]. Water has a comparatively higher surface tension/contact angle and, therefore, tends to trap vapor and form stable nucleation sites at lower superheats than wetting fluids. This translates into measured incipience superheats of
less than 1.5 °C for pool boiling of water from sintered porous surfaces [16,31].

The required superheat observed here of up to 19.8 °C for capillary-fed porous structures in vapor chambers contradicts the results for pool boiling. Altman et al. [26] performed device testing of complete vapor chambers that contain sintered powder wicks at sub-atmospheric pressure and observed incipience at superheats from 20 - 70 °C. Similarly, Liou et al. [32] tested sintered screen wick heat pipes and showed that heat fluxes up to 100 W/cm² did not induce boiling; wall superheats were not explicitly provided. Reduced saturation pressure alone is not a sufficient reason for the large discrepancy between capillary-fed and pool boiling results. Capillary-fed testing at atmospheric pressure in the current study using sintered powder wicks, and previously by Li and Peterson [2] using sintered screen wicks, both observed superheats on the order of 10 °C.

None of the previous studies provide further justification or propose a mechanism for sustained evaporation in capillary-fed wicks. Hsu’s model [33] for the required incipience superheat is based on a limiting thermal boundary layer thickness in the superheated liquid. It is widely used to understand the nucleation process and agrees with experimental trends with respect to subcooling, saturation pressure, and fluid properties. In the model, a threshold superheat required for incipience, independent of surface roughness, is proposed and is proportional to fluid properties and a limiting thermal layer thickness, $\delta_t$:

$$\Delta T_{\text{onset}} \propto \frac{\sigma_f T_{\text{sat}}}{\rho_l h_f \delta_t}$$  \hspace{1cm} (3)$$

In capillary-fed wicks, the liquid menisci recede and form a thin liquid film over the particle surfaces with increasing heat input. It may be postulated that these micron-scale liquid films form a thin thermal boundary layer that prevents bubble growth and increases the threshold superheat for nucleation compared to pool boiling, which forms a thicker thermal boundary layer under free convection. This is similar to the effect of bulk liquid convection on reducing $\delta_t$ and suppressing nucleation in flow boiling.

**CNT-coated monolithic samples.** Boiling curves for seven test runs using two CNT-coated samples are shown in Fig. 9b. The substrate superheat at incipience has a narrow range from 2.0 to 6.7 °C with a mean value of 3.7 °C and standard deviation of 1.6 °C. The transient temperature drop upon incipience exceeded 1 °C for only a single trial. The trials are highly repeatable for a given sample, but the performance difference between the two samples tested result in two boiling curve groupings that can be clearly observed in the figure.

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**Fig. 10** Regime map of heat flux versus superheat temperature for all steady-state data points acquired during the repeated capillary-fed incipience testing (Fig. 9a, Fig. 9b).

**Fig. 11** Comparison of a) relative cumulative frequency of superheat-dependent boiling incipience, and b) histogram of probability of incipience at a given substrate superheat for CNT-coated versus uncoated monolithic sintered copper powder.
The CNT-coated samples begin to boil at a lower superheat and over a narrower range than the uncoated samples. Fig. 11 provides a graphical performance comparison. The plot of relative cumulative frequency (Fig. 11a) depicts the percentage of total test trials for which onset of boiling began below a given superheat with a sampling interval of 2 °C. The slope provides insight into the distribution and predictability of incipience. The leftward shift and increased slope of the line corresponding to CNT-coated samples indicates that reduced incipience superheat and improved predictability are achieved with the CNT coating, respectively.

The incipience probability histogram in Fig. 11b plots the percentage of test trials during which boiling was observed in the 2 °C-wide x-axis intervals. In general, the distributions have a maximum at low superheats and gradually decrease with increasing superheat. One contradiction to this trend is the number of uncoated sample trials which began to boil above an 18 °C superheat. While it is difficult to speculate on the number of trials required to obtain a statistically significant dataset to represent all possible surfaces, it is likely that a sharp increase in incipience probability at high superheats is not a universal trend, and in this case the data is skewed by a single sample which tended to begin boiling at a higher superheat than average.

Nanostructured surfaces have been shown to reduce boiling incipience superheat for capillary-fed sintered powder wicks [26] but the mechanism for this enhancement is not well understood. Ujereh et al. [15] and McHale et al. [16] showed that CNT-coating of smooth copper and silicon surfaces reduces the incipience superheat required for pool boiling using FC-72 and water. In these cases, boiling occurs at a lower superheat because the nanotubes form microscale pores with a wide range of sizes – not available on the smooth surface – which are able to produce stable nucleation sites at lower superheats under Hsu’s theory for boiling incipience. Conventional theory also suggests that cavities formed in nanoscale pores within the CNT array would require immense superheats to become active due to the inverse relationship between cavity radius and required activation superheat [34], and therefore, likely do not participate.

The sintered powder wicks in the current study have numerous surface roughness features which create a wide range of potential microscale cavities, and yet, the CNT-coating reduces incipience. Therefore, the mechanism by which CNTs reduce the incipience superheat for capillary wicks may be attributed to: 1) an increase in the microscale thermal boundary layer (as occurs in flow boiling [35]), and/or 2) changes in the inner characteristics of the existing microscale cavities to reduce the required superheat. One such mechanism proposed by Li et al. [36] is the ability of a nanorod coating to increase the stability of a microcavity vapor embryo by feeding it with vapor trapped in the nanoscale pores. Similarly, any increase in the cavity angle, liquid contact angle, or change in shape of the microscale cavities within the sintered powder caused by the CNT coating could reduce the incipience superheat.

CONCLUSIONS

The implementation of passive phase-change cooling devices, such as vapor chambers, for applications which demand thermal management of high heat fluxes in confined geometries requires detailed investigation of incipience criteria and boiling performance of capillary-fed porous structures. Sintered copper powder wicks (200 µm thick), suitable for integration into sub-millimeter thickness vapor chambers, are evaluated in a capillary-fed boiling test facility using water as the working fluid. Without modification, the wicks sustain heat fluxes of up to 437 W/cm² at a superheat of 23 °C. Wick patterning and functionalized CNT-coating augmentation strategies are investigated. Unlike for thicker wicks, which benefit from the reduced vapor flow resistance created by patterning, the dryout superheat is reduced for 200 µm thick wicks upon patterning. However, a CNT array grown in the patterned recesses is observed to actively wick fluid and increase the dryout heat flux. A large incipience superheat overshoot, undesirable for device operation, is observed for capillary-fed sintered powder structures due to formation of thin liquid layers which can sustain evaporation at superheats greater than 10 °C. A detailed investigation of the superheat required for onset of boiling showed that the CNT-coating was able to reduce the mean surface superheat by 5.6 °C and standard deviation by 4.9 °C compared to uncoated samples.

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